

REMARKS

Applicants have amended their claims in order to further clarify the definition of various aspects of the present invention. Specifically, Applicants have amended claim 1 to recite that the pH is adjusted in a range of 6.0 to 9.0.

Applicants have added new claim 11 to the application, reciting that pH is adjusted in the range of 6.0 to 9.0 in the step of washing with water. In light of amendments to claim 1, and new claim 11, claim 2 has been canceled without prejudice or disclaimer. Moreover, dependencies of claims 3-6, 8 and 9 have been amended.

In addition to new claim 11, claims 12-14 have been added to the application. Claim 12, dependent on claim 1, defines a preferred range to which the pH is adjusted. Claim 13, dependent on claim 1, recites that the pH is adjusted by adding an alkali; and claim 14, dependent on claim 13, recites that this alkali is a same material as the basic catalyst.

As to the newly added claims, note, for example, page 13 of Applicants' specification.

Applicants respectfully submit that all of the claims presented for consideration by the Examiner patentably distinguish over the teachings of the references as applied by the Examiner in rejecting the claims as formerly in the application, that is, the teachings of the U.S. patents to Supplee, et al., No.

5,948,943, and to Palmer, et al., No. 3,956,406, under the provisions of 35 USC 103.

It is respectfully submitted that these references as applied by the Examiner would have neither taught nor would have suggested such a process for producing a polyol by reacting an aliphatic aldehyde, as in the present claims, including, inter alia, (a) a step of extraction which includes extracting the polyol from a concentrated reaction liquid with an extracting reagent, and (b) a step of washing with water which includes washing an extract liquid with water and separating the liquid into an oil layer containing the polyol and an aqueous layer, wherein an aliphatic aldehyde is represented by a specified formula is used as the extracting reagent, this extracting reagent being recovered after adjusting pH of the oil layer containing the polyol, the pH being adjusted in a range of 6.0 to 9.0. See claim 1.

More specifically, it is respectfully submitted that these references would have neither taught nor would have suggested such a process as in the present claims, wherein the pH is adjusted in the step of washing with water (see claim 11; note also claim 3); and/or wherein the pH is adjusted in the range of 6.5 to 8.0 (see claim 12).

Moreover, it is respectfully submitted that the teachings of the applied references would have neither taught nor would have suggested such a process

for producing a polyol as in the present claims, including, inter alia, wherein the pH is adjusted by adding an alkali (see claim 13); more specifically, wherein this alkali is a same material as the basic catalyst used as the catalyst in reacting the aliphatic aldehyde with formaldehyde (see claim 14; note also claim 3).

In addition, it is respectfully submitted that the teachings of the applied references would have neither disclosed nor would have suggested the other aspects of the present invention as in the remaining claims, including (but not limited to) wherein when the reacting agent is recovered from the oil layer, the oil layer is preliminarily heated in advance and flashed into an upper stage of a distillation column (note claim 4); and/or wherein water or steam is introduced into a bottom portion of a distillation column when recovering extracting reagent from the oil layer (see claim 5); and/or wherein a same aliphatic aldehyde as the aliphatic aldehyde used as the raw material of the reaction in forming the polyol, is used as the reacting reagent, with at least a portion of recovered extracting reagent used as the raw material (see claim 6); and/or washing of the extract liquid with water in the step of washing with water with removal and recycling of the extracting reagent as in claims 8 and 9.

The present invention is directed to a process for producing a polyol by reacting an aliphatic aldehyde with formaldehyde in the presence of a basic catalyst, and processing of the reaction product. In general, a process for

producing a polyol includes steps of reaction, extraction of the polyol from the reaction product liquid, separation of the extracting reagent and purification of the product by distillation. In the step of separation of the extracting reagent, the extracting reagent is separated from an extract liquid containing the polyol in, for example, a distillation process, with the obtained crude polyol being purified in a step of purification by distillation.

It has been proposed to utilize an aliphatic aldehyde as the extracting reagent; while such aliphatic aldehyde can be used in a continuous distillation of crude polyol, a great amount of acetals is formed from the polyol and the aldehyde used as the extracting reagent, in distillation under an atmospheric pressure for separating the extracting reagent, since aldehyde which is used as the extracting reagent is very reactive itself.

In addition, in previously proposed techniques the amount of the salts of formic acid remaining in the extract liquid is unduly large; such amount may be decreased by washing the extract liquid with water after the extraction. In this case, it is desirable that this washing water be removed in recycling the washing water to the step of extraction, however, concentration of water in the liquid to be treated by the extraction increases, and efficiency of the extraction decreases.

While reaction product liquid may be concentrated to prevent decrease in efficiency of the extraction, problems such as clogging of piping due to

separation of the salt of formic acid takes place, and operation becomes difficult.

On the other hand, when the washing water is recycled to the step of concentration of the reaction product with or without any treatment, degeneration of the extracting reagent and side reactions of the extracting reagent with the polyol take place.

Against this background, Applicants provide a technique for producing a polyol, wherein acetal and aldol compounds are formed only in small amounts and a high purity polyol is produced at a high yield, even when aliphatic aldehyde is used as the extracting reagent and it is reused for extraction. The present procedure, in various aspects, forms a high purity polyol which is efficiently separated from a salt of formic acid, while accumulation of impurities in an extracting reagent is prevented, and wherein the polyol and a salt of formic acid are efficiently separated by a stable operation.

The foregoing objectives are achieved according to various aspects of the present invention. As a first aspect, the extracting reagent is recovered after adjusting the pH of the oil layer containing the polyol which is separated in the step of washing with water. Since the reaction product liquid is extracted with a specific aliphatic aldehyde used as the extracting reagent and the extracting reagent is recovered after pH of the extract liquid is adjusted, continuous distillation can be conducted with suppressed formation of byproducts and high

purity polyol can be obtained. Note the last full paragraph on page 17 of Applicants' specification.

As another aspect of the present invention, a same aliphatic aldehyde, as the aliphatic aldehyde used as the raw material in forming the polyol, is used as the extracting reagent, and the recovered extracting reagent is recycled, e.g., to the reaction. Through this aspect of the present invention, accumulation of impurities (aldols, methanol, alkenals and the like) in the extracting reagent is suppressed without adverse effects on the result of the reaction, and the polyol and salt of formic acid can be efficiently separated from each other. See the paragraph bridging pages 17 and 18 of Applicants' specification.

According to a further aspect of the present invention, in recovering/removing extracting reagents, the extracting reagent is recycled to the extractor. According thereto, an efficient extraction can be achieved at a high yield of extraction of the polyol and a high fraction of the removed salt of formic acid in the step of extraction, purification by continuous distillation can be conducted, and high quality polyol can be obtained with stability. Note the first full paragraph on page 18 of Applicants' specification.

Supplee, et al. discloses production of low color crude trimethylolpropane (TMP). In the disclosed process, the TMP is prepared by condensation of n-butyraldehyde and formaldehyde in an alkali solution, the mixture then being

placed through an extractor. This patent discloses that a high concentration of low color crude TMP may be obtained by taking a stream of hot solvent/water/TMP from the extractor and allowing the mixture, with or without additional water, to cool and phase separate. Upon separation of the phases, TMP generally having an acid wash of less than 5 is recovered from the aqueous phase. See column 2, lines 2-14. Note also column 2, lines 49-57.

As can be seen from the foregoing, as well as from a full review of Supplee, et al., it is respectfully submitted that this patent does not disclose, nor would have suggested, the adjusting of pH, much less the advantages achieved thereby, as in the present invention. By adjusting pH of the oil layer, as in the present invention, a high purity polyol is obtained at high yield, because formation of acetal compound and aldol compounds is suppressed. The effect of adjusting the pH of the oil layer is clearly seen in comparing Example 1 and Comparative Example 1 of the present application, on pages 18-22 of Applicants' specification. In Example 1, the amount of acetal in the residual liquid in the column was 0.24% based on the amount of NBAL (n-butyraldehyde) used as the raw material. In Comparative Example 1, the amount of acetal in the residual liquid in the column was 15.3% based on the amount of NBAL used as the raw material. As seen in these examples, a high purity polyol is obtained at high yield by adjusting pH of the oil layer, as in the present

invention.

In addition, it is emphasized that Supplee, et al. is concerned with acquiring crude trimethylolpropane of low reacted color, and would have neither taught nor would have suggested the other aspects of the present invention, utilizing the specified aliphatic aldehyde as the extracting reagent, and with recycle as discussed previously.

It is respectfully submitted that the additional teachings of Palmer, et al. would not have rectified the deficiencies of Supplee, et al., such that the present invention as a whole would have been obvious to one of ordinary skill in the art.

Palmer, et al. discloses a technique for purification of trimethylolpropane (TMP), wherein the aqueous alkaline processed stream from the condensation of n-butyraldehyde and formaldehyde is extracted with a first solvent and the resulting mixture of TMP and the first solvent is combined with a second solvent, which second solvent serves to further reduce the aqueous alkaline content of the extract. See column 1, lines 53-59. This first solvent is described in the paragraph bridging columns 1 and 2 of this patent, and includes lower boiling aliphatic alcohols. The second solvent must be substantially immiscible with water and easily separated from the first solvent and trimethylolpropane, examples including, e.g., toluene, xylene, and others. See column 2, lines 5-21.

It is respectfully submitted that Palmer, et al. discloses use of sequential

solvents for purification of TMP by extraction of TMP from the process effluent.

Even assuming, arguendo, that the teachings of Palmer, et al. were properly combinable with the teachings of Supplee, et al., such combined teachings would have neither taught nor would have suggested the presently claimed subject matter, including adjustment of pH, particularly adjustment in a range of 6.0 to 9.0; use of the aliphatic aldehyde as extracting reagent, and recycle, as in various aspects of the present invention and set forth in the present claims. In this regard, it is emphasized that Palmer, et al. utilizes a sequence of two different solvents for the extraction, the first one being a lower boiling aliphatic alcohol and the second being illustrated, for example, in column 2, lines 18-21; and that this patent does not disclose nor would have suggested, use of the aliphatic aldehyde as extracting agent, and advantages achieved thereby, as in the present invention and discussed previously.

In view of the foregoing comments and amendments, reconsideration and allowance of all claims remaining in the application are respectfully requested.

Attached hereto is a marked-up version of the changes made in the claims by the current Amendment. This marked-up version is on the attached pages, the first page of which is captioned "VERSION WITH MARKINGS TO SHOW CHANGES MADE".

To the extent necessary, Applicants petition for an extension of time under

37 CFR § 1.136. Please charge any shortage in fees due in connection with the filing of this paper, including extension of time fees, to the Deposit Account No. 01-2135 (Case No. 396.40193X00) and please credit any excess fees to such Deposit Account.

Respectfully submitted,

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A handwritten signature in black ink, appearing to read 'William I. Solomon', with a long horizontal flourish extending to the right.

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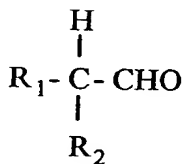
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VERSION WITH MARKINGS TO SHOW CHANGES MADE

IN THE CLAIMS

Please cancel claim 2 without prejudice or disclaimer, and amend the claims remaining in the application as follows:

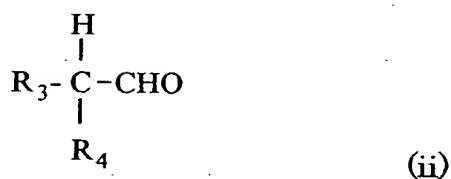
1. (Amended) A process for producing a polyol by reacting an aliphatic aldehyde represented by formula (i):



(i)

wherein R₁ and R₂ each represent hydrogen atom or an aliphatic alkyl group having 1 to 6 carbon atoms, with formaldehyde in a presence of a basic catalyst, which process comprises (1) a step of concentration which comprises removing water and unreacted formaldehyde from a reaction liquid by distillation; (2) a step of extraction which comprises extracting the polyol from a concentrated reaction liquid with an extracting reagent; and (3) a step of washing with water which comprises washing an extract liquid with water and separating the liquid into an oil layer containing the polyol and an aqueous layer; wherein an aliphatic

aldehyde represented by formula (ii):



wherein R₃ represents hydrogen atom or an aliphatic alkyl group having 1 or 2 carbon atoms and R₄ represents an aliphatic alkyl group having 1 to 5 carbon atoms is used as the extracting reagent, and the extracting reagent is recovered after adjusting pH of the oil layer containing the polyol which is separated in the step of washing with water, the pH being adjusted in a range of 6.0 to 9.0.

3. (Amended) A process for producing a polyol according to Claim [1] 11, wherein the basic catalyst is used for adjusting pH in the step of washing with water.

4. (Amended) A process for producing a polyol according to Claim [1] 11, wherein, when the extracting reagent is recovered from the oil layer, the oil layer is preliminarily heated in advance and flashed into an upper stage of a distillation column.

5. (Amended) A process for producing a polyol according to Claim [1] 11, wherein the extracting reagent is recovered from the oil layer while water or steam is introduced into a bottom portion of a distillation column.

6. (Amended) A process for producing a polyol according to Claim [1] 11, wherein a same aliphatic aldehyde as the aliphatic aldehyde used as a raw material of the reaction is used as the extracting reagent and at least a portion of the recovered extracting reagent is used as the raw material.

8. (Twice Amended) A process for producing a polyol according to Claim [1] 11, wherein the extract liquid is washed with water in the step of washing with water, the extracting reagent in a separated aqueous layer using a decanter is removed by distillation and water obtained from a bottom of a distillation column in the distillation is recycled to the step of concentration.

9. (Twice Amended) A process for producing a polyol according to Claim [1] 11, wherein the extract liquid is washed with water in the step of washing with water, the extracting reagent and a portion of water in a separated aqueous layer using a decanter are removed by distillation and a liquid obtained

from a bottom of a distillation column in the distillation is recycled to the step of extraction.